

Customer No. 20350
TOWNSEND and TOWNSEND and CREW LLP
(650) 326-2400

Attorney Docket No. AM888/T0020-1

ASSISTANT COMMISSIONER FOR PATENTS
BOX PATENT APPLICATION
Washington, D.C. 20231

"Express Mail" Label No. EM 03514867345
Date of Deposit: November 12, 1998

I hereby certify that this is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service under 37 CFR 1.10 on the date indicated above, addressed to

Assistant Commissioner for Patents
Washington, D.C. 20231

By: Scott W. Hewett

11/12/98
19096
U.S. PTO
JCS51

Sir:

Transmitted herewith for filing is the

- ☐ patent application of
☒ continuation patent application of
☐ divisional patent application of
☐ continuation-in-part patent application of

Inventor(s)/Applicant Identifier: NGUYEN et al.

For: LIQUID PHOSPHOROUS PRECURSOR DELIVERY APPARATUS

☒ This application claims priority from each of the following Application Nos./filing dates:

08/568,193 filed 12/06/95

the disclosure(s) of which is (are) incorporated by reference.

☒ Please amend this application by adding the following before the first sentence: "This application is a ☐ continuation ☐ continuation-in-part of and claims the benefit of U.S. Provisional Application No. 60/_____, filed _____, the disclosure of which is incorporated by reference."

Enclosed are:

- ☒ 2 sheet(s) of ☒ formal ☐ informal drawing(s), 6 pages of specification including 6 pages of claims, abstract, and title page.
☒ A copy of the prior signed Declaration & Power of Attorney.
☒ A petition to extend time to respond in the parent application.
☒ Preliminary Amendment.

(Col. 1)

(Col. 2)

SMALL ENTITY

OTHER THAN
SMALL ENTITY

FOR:	NO. FILED		NO. EXTRA	
BASIC FEE				
TOTAL CLAIMS	25	- 20	=	*5
INDEP. CLAIMS	4	- 3	=	*1
<input type="checkbox"/> MULTIPLE DEPENDENT CLAIM PRESENTED				

RATE	FEE
	\$395.00
x \$11.00 =	
x \$41.00 =	
+ \$135.00 =	
TOTAL	

RATE	FEE
	\$790.00
x \$22.00 =	\$110.00
x \$82.00 =	\$82.00
+ \$270.00 =	
TOTAL	\$982.00

* If the difference in Col. 1 is less than 0, enter "0" in Col. 2.

Please charge Deposit Account No. 20-1430 as follows:

- ☒ Filing fee
☒ Any additional fees associated with this paper or during the pendency of this application.

\$ \$982.00

2 extra copies of this sheet are enclosed.

Telephone: Facsimile:
(650) 326-2400 (650) 326-2422

Respectfully submitted,

TOWNSEND and TOWNSEND and CREW LLP

Scott W. Hewett

Scott W. Hewett
Reg No.: 41,836
Attorneys for Applicant

I hereby certify that this correspondence is being deposited with the United States Postal Service as first class mail in an envelope addressed to:

PATENT
Attorney Docket No.:AM888/T20.10

Assistant Commissioner for Patents
Washington, D.C. 20231

On Nov. 12, 1998

TOWNSEND and TOWNSEND and CREW LLP

By: Scott Hewitt

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of:

NGUYEN et al.

Application No.: Unassigned

Filed: Herewith

For: LIQUID PHOSPHOROUS
PRECURSOR DELIVERY APPARATU

Art Unit: Unassigned

PRELIMINARY AMENDMENT

Assistant Commissioner for Patents
Washington, D.C. 20231

Sir:

Prior to the examination of the above-referenced application on the merits, please enter the following amendments and additions to the claims.

IN THE CLAIMS:

Please amend claims 1-4, 8-11, 15, 16, 20-22, and 28, cancel claims 31-45, and add claim 46 as follows:

- 1 1. (AMENDED) An apparatus for use with a liquid phosphorous precursor
- 2 compound comprising:
- 3 a container adapted to hold the liquid phosphorous precursor
- 4 compound;
- 5 a conduit; and

Application No.:

Page 2

an orifice disposed between the liquid container and the conduit, wherein at least one of the liquid container, the orifice, and the conduit has a surface of a **[metal] stainless steel** alloy having less than 5 % nickel.

2. (AMENDED) The apparatus of claim 1 wherein said **[metal] stainless steel** alloy **[comprises stainless steel alloy with]** has at least 15% chromium.

3. (AMENDED) The apparatus of claim 1 wherein said **[metal] stainless steel** has alloy **[comprises a stainless steel having]** has less than 1% nickel.

4. (AMENDED) The apparatus of claim 1 wherein said **[metal] stainless steel** alloy is selected from the group consisting of stainless steel alloy 430, stainless steel alloy 440, and stainless steel alloy 446.

8. (AMENDED) An apparatus for delivering a liquid phosphorous precursor compound, comprising:

a container adapted to hold said liquid phosphorous precursor compound;

a conduit configured to convey said liquid phosphorous precursor compound or a gaseous product of said liquid phosphorous precursor compound from the container;

a heating surface coupled to at least one of a portion of said container and a portion of said conduit;

wherein at least one of said portion of said container and said portion of said conduit is composed of **[an] a stainless steel** alloy having less than 5 percent nickel.

9. (AMENDED) The apparatus of claim 8 wherein said **[metal alloy comprises]** stainless steel alloy **[with] comprises** at least 15% chromium.

10. (AMENDED) The apparatus of claim 8 wherein said **[metal] stainless steel** alloy comprises **[stainless steel having]** less than 1% nickel.

11. (AMENDED) The apparatus of claim 8 wherein said **[metal]** stainless steel alloy is selected from the group consisting of stainless steel alloy 430, stainless steel alloy 440, and stainless steel alloy 446.

15. (AMENDED) The apparatus of claim 8 wherein said apparatus comprises an injection system for delivering gases to a chemical reaction chamber for semiconductor wafer fabrication, and wherein said injection system includes an injection valve composed of **[an]** a stainless steel alloy having less than 5 percent nickel.

16. (AMENDED) The apparatus of claim 8 wherein said portion composed of **[an]** the stainless steel alloy comprises a gasket and a seal.

20. (AMENDED) A liquid flow injection valve for supplying TEPO, TMP or TEP to a chemical vapor deposition (CVD) chamber comprising:
an injection orifice for connecting to a source of liquid TEPO, TMP or TEP; and
a valve outlet for delivering a gaseous mixture generated from said liquid TEPO, TMP or TEP to said CVD chamber;
said injection orifice including a **[metal]** stainless steel alloy having less than 5% nickel.

21. (AMENDED) The valve of claim 20 wherein said **[metal comprises a]** stainless steel alloy **[with]** has at least 15% chromium.

22. (AMENDED) The valve of claim 20 wherein said **[metal]** stainless steel alloy is selected from the group consisting of stainless steel alloy 430, stainless steel alloy 440, and stainless steel alloy 446.

28. (AMENDED) A method for injecting gaseous phosphorous precursor into a **[CVD]** chemical vapor deposition chamber, the method comprising **[the steps of]**:

Application No.:

Page 4

3 providing a liquid TEPO, TMP or TEP through an injection valve including a
4 [metal] stainless steel alloy having less than 10% nickel;
5 providing a carrier gas through said valve;
6 creating a pressure differential in said valve; and
7 heating said injection valve.

1 31.-45. [CANCELED]

1 46. (NEW) An apparatus for use with a liquid phosphorous precursor
2 compound, the apparatus comprising:

3
4 a container adapted to hold the liquid phosphorous precursor compound;
5 a conduit; and
6 an orifice disposed between the liquid container and the conduit, wherein at
7 least one of the liquid container, the orifice, and the conduit has a surface of a stainless steel
8 alloy having less than 10 % nickel.

REMARKS

Claims 1-4, 8-11, 15, 16, 20-22, and 28 have been amended to specifically recite a stainless steel alloy, as recommended by the Examiner in the telephone conference of November 12, 1998. In that telephone conference, the Examiner indicated that claims 31-45 would be allowed in the parent case, for which the Applicants sincerely thank the Examiner. Accordingly, claims 31-45 have been canceled. Thus, claims 1-30 and 46 are pending.

CONCLUSION

In view of the foregoing, Applicants believe all claims now pending in this Application are in condition for allowance. Examination of all claims and the issuance of a

Application No.:

Page 5

formal Notice of Allowance at an early date is respectfully requested.

If the Examiner believes a telephone conference would expedite prosecution of this application, please telephone the undersigned at 650-326-2400.

Respectfully submitted,



Scott W. Hewett
Reg. No. 41,836

TOWNSEND and TOWNSEND and CREW LLP

Tel: (650) 326-2400

Fax: (650) 326-2422

SWH:sgk

PA 160479 v1

0910061-11293

PATENT APPLICATION

LIQUID PHOSPHOROUS PRECURSOR DELIVERY APPARATUS

Inventors:

Chau Nguyen, a citizen of the United States, residing
at 1748 Morrill Avenue, San Jose, CA 95132

Visweswaren Sivaramakrishnan, a citizen of India, residing at
20303 N. Cove Square, Cupertino, CA 95014

Assignee:

Applied Materials, Inc.
P.O. Box 58039
3105 Kifer Road M/S 2634
Santa Clara, CA 95052-8039

Entity:

Large

TOWNSEND and TOWNSEND and CREW
Steuart Street Tower, 20th Floor
One Market Plaza
San Francisco, California 94105
(415) 543-9600

LIQUID PHOSPHOROUS PRECURSOR DELIVERY APPARATUS

BACKGROUND OF THE INVENTION

The present invention relates to delivery systems for liquid phosphorous precursors, and in particular to stainless steel containers, piping and injection valves for injecting liquid triethylphosphate (TEPO), TMP or TEP into a chemical vapor deposition (CVD) chamber.

A variety of different systems can be used to deliver processing gases to a chemical vapor reaction chamber. In a boiler system, the liquid is heated into vapor form. In a "bubbler" system, gaseous helium is introduced into a liquid in a container, resulting in some of the liquid being bubbled out of solution. When the liquid contains a phosphorous precursor, such as TEPO, TMP or TEP, and the container or piping is stainless steel, residue build-up has been observed, in particular where the stainless steel is exposed to heat.

Injection valves are often used for providing a processing gas to a CVD chamber. In one method of doing this, the active gas component is provided in liquid form to an injection valve. The injection valve provides the liquid through an orifice past which a carrier gas is provided. A pressure drop is created which causes the liquid to vaporize into gaseous form. Typically, a heater is also provided on the valve to prevent condensation of the processing gas. A typical inert carrier gas is helium.

One problem encountered with such valves is the build-up of residue around the orifice, which can prevent proper seating of a cut-off plug to hinder control of the valve. Excessive build-up of residue can also block the orifice itself, or severely restrict the flow of liquid through the orifice. Residue build-up on other surfaces can contaminate subsequent gases flowing across the surface or contained in the container.

Accordingly, it would be desirable to have an liquid phosphorous precursor delivery system which minimizes the build-up of residue on stainless steel surfaces.

SUMMARY OF THE INVENTION

The present invention recognizes that the build-up of residue in a metal alloy injection valve used to inject a liquid phosphorous precursor compound is due to the nickel in the alloy affecting the liquid phosphorous precursor compound. The invention provides components manufactured of an alloy having a low nickel content, preferably less than 5% nickel, and more preferably less than 1%. In an additional aspect of the invention, the alloy is provided with a higher chromium content, preferably at least 15% chromium, more preferably 16-27%.

The chromium appears to inhibit the leaching of the metal by the liquid phosphorous precursor compound, thus preventing the nickel being leached out of the metal to affect the liquid phosphorous precursor compound. The nickel appears to act as a catalyst for causing decomposition of the phosphorous precursor compound when heated. Preferably, the components exposed to the phosphorous precursor compound and heat are made of stainless steel alloys of standard industrial designations 430, 440, or 446, which all have a nickel content of less than 1%.

In one embodiment, an injection valve is made of stainless steel alloys of standard industrial designations 430, 440, or 446. This alloy is preferably used for the body of the valve, but in particular for at least the portions of the valve around the injection orifice.

In another embodiment, a polyamide is used for a plug in an injection valve instead of prior art fluoropolymers. The polyamide, preferably Vespel (a 3M product) is used, and exhibits better tolerance to the liquid phosphorous precursor compound and heat. The polyamide can also be used for gaskets and seals.

For a fuller understanding of the nature and advantages of the invention, reference should be made to the ensuing detailed description taken in conjunction with the accompanying drawings.

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is a block diagram of a liquid injection system including an injection valve according to the present invention;

Fig. 2 is a block diagram showing an injection system having multiple injection valves, including an injection valve for TEPO according to the present invention;

Fig. 3 is a detailed diagram of the injection valve according to the present invention; and

Figs. 4 and 5 are diagrams illustrating the build-up of residue on an injection valve.

DESCRIPTION OF THE PREFERRED EMBODIMENT

The following description illustrates one embodiment of the present invention using an injection valve system. The invention also applies to boiler and bubbler systems, with the low nickel stainless steel alloy being used for liquid containers in such systems, or for tubing or conduit, or for any other portion that is exposed to a liquid phosphorous precursor containing compound and also to heat.

Fig. 1 illustrates a basic injection valve system for providing process gases to a process chamber 12. A liquid container 14 containing liquid TEPO 16 is pressurized using helium provided through a valve 18. The pressurized helium in the top of container 14 forces the liquid TEPO 16 through a line 20 to a liquid mass flow meter (LFM) 22 which meters the amount of liquid provided to an injection valve 24 via an injection line 26. The injection valve is also provided with a carrier gas, preferably helium, through a mass flow controller (MFC) 28 and a carrier gas injection line 30. Injection valve 24 converts the liquid from injection line 26 into gaseous form, and provides it along with the carrier gas through an outlet line 32 to process chamber 12. Process chamber 12 includes a monitoring pressure sensor 34 and a vacuum pump 36 for removing exhaust gases.

Fig. 2 illustrates the application of multiple gases to chamber 12, including the liquid TEPO provided through injection valve 24. In Fig. 2, there is also shown a control valve 38 for liquid TEPO connected to a control valve 44 allowing purging of the gas lines with nitrogen (N_2).

Fig. 2 shows injection valve 24 being connected to a common gas line 42 connected to process chamber 12. Also included in gas line 42 are an injection valve 44 for liquid TEOS and an injection valve 46 for liquid TEB. Injection valve 44 has associated with it a liquid flow meter 48 and valves 50 and 52 for controlling the liquid TEOS and nitrogen purge. A degasser 51 may optionally be included to remove helium, where helium is used to pressurize the TEOS (degassers may be used in other gas lines as well). Similarly, injection valve 46 is connected to a liquid flow meter 54 and associated

valves 56 and 58 for controlling the liquid TEB and nitrogen. Finally, mass flow controllers 60 and 62 connect to gas line 42 providing a low flow carrier, and high flow carrier, respectively. Preferably, helium is typically used as the carrier.

Fig. 3 shows injection valve 24 in more detail. The TEPO liquid is provided through liquid mass flow meter 22 via inlet line 26. The inlet line is connected to a chamber 64 which includes a spring 66 for biasing against a plug 68. Plug 68 is moved in and out under processor control in order to control the amount of flow of liquid TEPO. The top of chamber 64 is a gas orifice 70.

Helium is provided as a carrier gas through an inlet line 30, and the combination gas mixture is provided through an outlet line 32 to the process chamber. The gas flow of the helium over the orifice causes a pressure drop which causes the liquid TEPO to vaporize, and be carried with the helium through outlet line 32 to the process chamber. Necessarily, orifice 70 is small in order to aid this vaporization process, and thus is vulnerable to residue build-up. Prior art valves typically include a valve body, including the portion surrounding the orifice, made of a stainless steel alloy. For example, stainless steel alloy SST 316 is used in prior art valves manufactured by Lintec of Japan.

Plug 68 in existing valves is a compressible sealer typically made of Kel-F (a 3M fluoropolymer). We have found that Kel-F tends to swell up and break. Accordingly, another aspect of the present invention is the use of Vespel (DuPont polyamide resin) for the plug. Vespel can also be used for gaskets and seals in any system which utilizes a liquid phosphorous precursor compound.

The valve also includes a shut-off plug 72 which can be lowered to close the orifice when flow is desired to be shut off. Plug 72 is also preferably made of Vespel. Also included are heater elements 74 which function to heat the valve to prevent condensation of the gaseous mixture. A thermal couple 76 allows monitoring of the temperature of the valve.

Fig. 4 illustrates a residue build-up 80 around orifice 70 to a level of 300μ . This build-up does not substantially affect the flow of gas out of the orifice, but does impact the proper seating of shut-off plug 72 when it is desirable to stop the flow of TEPO.

Fig. 5 illustrates a build-up of residue to a thickness of 1800μ , which clogs the orifice itself, as shown by residue 82 in Fig. 5. As can be seen, orifice 70 is completely clogged at this point. Typically, the orifice itself has a diameter of 2 mm.

The inventors of the present invention determined through a series of tests that the presence of nickel in the stainless steel alloy of the valve around orifice 70 was affecting the liquid TEPO, causing the residue build-up. The prior art valves using the stainless steel alloy of SST 316 would typically contain approximately 12-15% nickel, and 16-18% chromium. In an experiment, a valve made of a stainless steel alloy 430, which contains approximately 0.15% nickel and 16-18% chromium was used. The use of such a valve allowed TEPO to flow for 189 hours (equivalent to a throughput of 11,300 wafers). The prior art valve using the 316 alloy, on the other hand, has been typically observed to have a throughput of 1800 wafers prior to clogging due to residue build-up. On the other hand, the 430 test still had no significant residue build-up after 189 hours, suggesting that a much longer lifetime was still available to the valve. The build-up of the residue which has been observed may be due to the nickel helping to decompose TEPO into phosphoric acid and ethanol. This can be avoided by limiting the amount of nickel in the alloy. In addition, the presence of chromium inhibits the leaching of the nickel out of the metal by the TEPO liquid. Alloys with a higher chromium content are preferred, but may be more expensive. Alloy 446, for instance, has approximately 0.6% nickel and 23-27% chromium. Alloy 440 has 0.6% nickel and 16-18% chromium.

In addition, by empirical observation, it was determined that a temperature of approximately $160-170^{\circ}\text{C}$, preferably 165°C , for the valve provided an optimum flow of the TEPO liquid, avoiding residue build-up.

The TEPO liquid, used for generating phosphorous precursor gas, is typically used for the BPSG (Boronphosphosilicate glass) and PSG (phosphosilicate glass) process steps in the processing of a wafer.

As will be understood by those with skill in the art, the present invention may be embodied in other specific forms without departing from the spirit or essential characteristics thereof. For example, stainless steel alloys having a higher chromium content or lower nickel content could be used. Additionally, the valve could have an appropriate alloy of stainless steel only around the sensitive orifice area of the valve. The low nickel stainless steel alloy could also be used for stainless steel gaskets. Alternately, a different type of processing system, such as a distillation system with a

WHAT IS CLAIMED IS:

- 1 1. An apparatus comprising:
2 a metal alloy surface in contact with a liquid phosphorous precursor
3 compound;
4 said metal alloy having less than 5% nickel.
- 1 2. The apparatus of claim 1 wherein said metal alloy is a stainless steel
2 alloy with at least 15% chromium.
- 1 3. The apparatus of claim 1 wherein said metal alloy is stainless steel
2 having less than 1% nickel.
- 1 4. The apparatus of claim 1 wherein said metal alloy is one of stainless
2 steel alloys 430, 440 and 446.
- 1 5. The apparatus of claim 1 wherein said phosphorous precursor
2 compound is TEPO, TMP or TEP.
- 1 6. The apparatus of claim 1 wherein said phosphorous precursor is TMP.
- 1 7. The apparatus of claim 1 wherein said phosphorous precursor
2 compound is TEP.
- 1 8. An apparatus for delivering a liquid phosphorous precursor compound,
2 comprising:
3 a container for holding said liquid phosphorous precursor
4 compound;
5 a conduit for delivering said liquid phosphorous precursor
6 compound or a gaseous product of said liquid phosphorous precursor
7 compound;
8 a heating surface coupled to at least one of said container and a
9 portion of said conduit;

10 wherein at least a portion of said container or said conduit is
11 composed of an alloy having less than 5 percent nickel.

1 9. The apparatus of claim 8 wherein said metal alloy is a stainless steel
2 alloy with at least 15% chromium.

1 10. The apparatus of claim 8 wherein said metal alloy is stainless steel
2 having less than 1% nickel.

1 11. The apparatus of claim 8 wherein said metal alloy is one of stainless
2 steel alloys 430, 440 and 446.

1 12. The apparatus of claim 8 further comprising a heater for heating said
2 heating surface to a temperature of 160-170 degrees Celsius.

1 13. The apparatus of claim 8 wherein said apparatus is a bubbler system
2 for delivering gases to a chemical reaction chamber for semiconductor wafers.

1 14. The apparatus of claim 8 wherein said apparatus is a boiler system for
2 delivering gases to a chemical reaction chamber for semiconductor wafers.

1 15. The apparatus of claim 8 wherein said apparatus is an injection system
2 for delivering gases to a chemical reaction chamber for semiconductor wafers, and
3 wherein said portion composed of an alloy is an injection valve.

1 16. The apparatus of claim 8 wherein said portion composed of an alloy is
2 one of a gasket and a seal.

1 17. The apparatus of claim 8 wherein said phosphorous precursor
2 compound is TEPO, TMP or TEP.

1 18. The apparatus of claim 8 wherein said phosphorous precursor is TMP.

1 19. The apparatus of claim 8 wherein said phosphorous precursor
2 compound is TEP.

1 20. A liquid flow injection valve for supplying TEPO, TMP or TEP to a
2 chemical vapor deposition (CVD) chamber comprising:

3 an injection orifice for connecting to a source of liquid TEPO,
4 TMP or TEP; and

5 a valve outlet for delivering a gaseous mixture generated from said
6 liquid TEPO, TMP or TEP to said CVD chamber;

7 said injection orifice including a metal alloy having less than 5%
8 nickel.

21. The valve of claim 20 wherein said metal is a stainless steel alloy with
at least 15% chromium.

22. The valve of claim 20 wherein said metal is one of stainless steel
alloys 430, 440 and 446.

23. The valve of claim 20 further comprising a heater for heating said
valve to a temperature of 160-170 degrees Celsius.

1 24. The valve of claim 20 further comprising a plug in said valve
2 composed of a polyamide.

1 25. The valve of claim 24 wherein said polyamide is Vespel.

1 26. A liquid injection system for a CVD chamber comprising:

2 a container for holding liquid TEPO, TMP or TEP;

3 an injection valve for converting said liquid TEPO, TMP or TEP
4 into gaseous form, said injection valve having portions in contact with said
5 liquid TEPO, TMP or TEP composed of a stainless steel alloy having less
6 than 5% nickel and at least 15% chromium;

7 a liquid TEPO, TMP or TEP injection line coupling said container
8 to said injection valve;
9 a carrier gas source line coupled to said injection valve; and
10 an outlet line coupling said injection valve to said CVD chamber.

1 27. The system of claim 26 wherein said stainless steel alloy is one of
2 stainless steel alloys 430, 440 and 446.

1 28. A method for injecting gaseous phosphorous precursor into a CVD
2 chamber comprising the steps of:
3 providing a liquid TEPO, TMP or TEP through an injection valve
4 including a metal alloy having less than 10% nickel;
5 providing a carrier gas through said valve;
6 creating a pressure differential in said valve; and
7 heating said injection valve.

1 29. The method of claim 28 further comprising the step of heating said
2 valve to a temperature of 160-170 degrees Celsius.

1 30. The method of claim 29 wherein said valve is heated to approximately
2 165 degrees Celsius.

1 31. An apparatus comprising:
2 a sealer in contact with a liquid phosphorous precursor compound;
3 said sealer being a polyamide.

1 32. The apparatus of claim 31 wherein said phosphorous precursor
2 compound is one of TEPO, TMP or TEP.

1 33. The apparatus of claim 31 wherein said sealer is a shut-off or control
2 plug in a valve.

1 34. The apparatus of claim 31 wherein said sealer is a gasket.

1 35. An apparatus for delivering a liquid phosphorous precursor
2 compound, comprising:

3 a container for holding said liquid phosphorous precursor
4 compound;

5 a conduit for delivering said liquid phosphorous precursor
6 compound or a gaseous product of said liquid phosphorous precursor
7 compound;

8 wherein at least a portion of said container or said conduit includes
9 a sealer composed of a polyamide.

1 36. The apparatus of claim 35 wherein said sealer is a shut-off or control
2 plug in a valve.

3 37. The apparatus of claim 35 wherein said polyamide is Vespel.

4 38. The apparatus of claim 35 wherein said apparatus is a bubbler system
5 for delivering gases to a chemical reaction chamber for semiconductor wafers.

6 39. The apparatus of claim 35 wherein said apparatus is a boiler system
7 for delivering gases to a chemical reaction chamber for semiconductor wafers.

8 40. The apparatus of claim 35 wherein said apparatus is an injection
9 system for delivering gases to a chemical reaction chamber for semiconductor wafers,
1 and wherein said sealer is a plug in an injection valve.

2 41. The apparatus of claim 35 wherein said sealer is a gasket.

3 42. The apparatus of claim 35 wherein said phosphorous precursor
4 compound is one of TEPO, TMP or TEP.

5 43. The apparatus of claim 35 wherein a portion of said container or said
6 conduit is composed of a stainless steel alloy having less than 5% nickel.

1 44. An liquid flow injection valve for supplying a liquid phosphorous
2 precursor source to a chemical vapor deposition (CVD) chamber comprising:
3 a container of said liquid phosphorous precursor, said liquid
4 phosphorous precursor being one of TEPO, TMP or TEP:
5 an injection orifice for connecting to said container; and
6 a valve outlet for delivering a gaseous mixture generated from said
7 liquid phosphorous precursor compound to said CVD chamber;
8 a shut-off or control plug in said valve, said plug being composed
9 of Vespel.

1 45. A liquid injection system for a CVD chamber comprising:
2 a container for holding liquid phosphorous precursor compound,
3 said liquid phosphorous precursor compound being one of TEPO, TMP or
4 TEP;
5 an injection valve for converting said liquid phosphorous precursor
6 into gaseous form, said injection valve having portions in contact with said
7 liquid phosphorous precursor compound composed of a stainless steel alloy
8 having less than 5% nickel and at least 15% chromium;
9 a shut-off or control plug in said injection valve, said plug being
10 composed of a polyamide;
11 a liquid phosphorous precursor compound injection line coupling
12 said container to said injection valve;
13 a carrier gas source line coupled to said injection valve; and
14 an outlet line coupling said injection valve to said CVD chamber.

LIQUID PHOSPHOROUS PRECURSOR DELIVERY APPARATUS

ABSTRACT OF THE DISCLOSURE

5 The present invention recognizes that the build-up of residue in a metal
alloy injection valve used to inject a liquid phosphorous precursor compound is due to the
nickel in the alloy affecting the liquid phosphorous precursor compound. The invention
thus provides components manufactured of an alloy having a low nickel content,
preferably less than 5% nickel, and more preferably less than 1%. In an additional
10 aspect of the invention, the alloy is provided with a higher chromium content, preferably
at least 15% chromium, more preferably 16-27%.

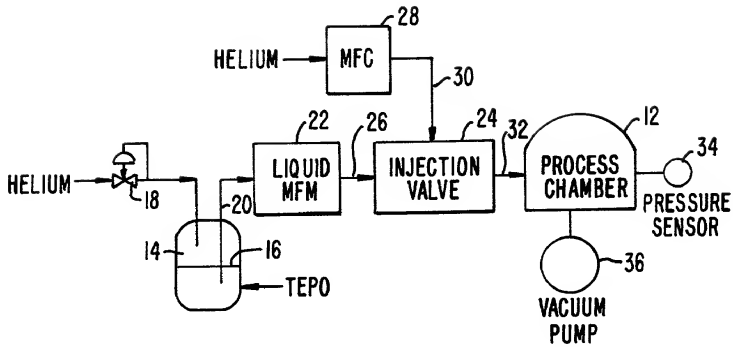


FIG. 1.

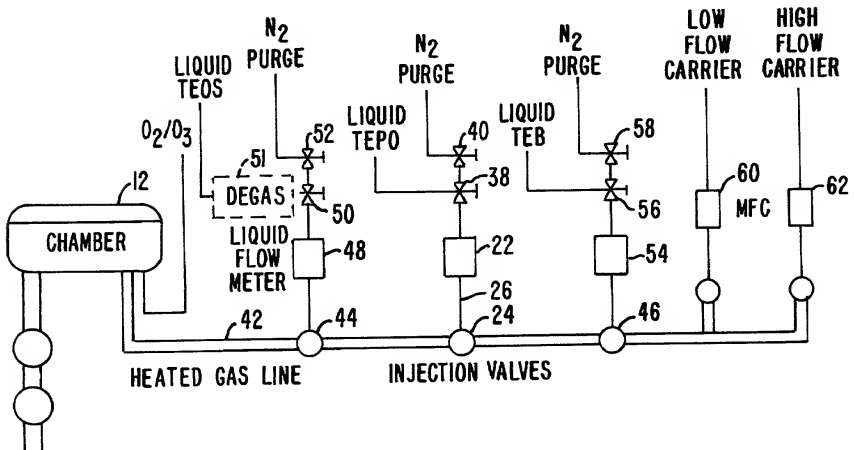
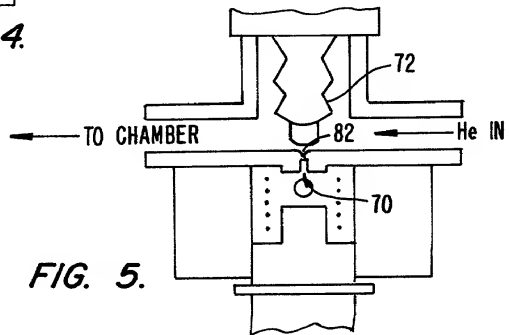
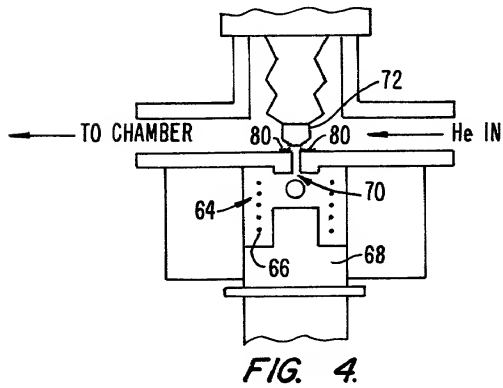
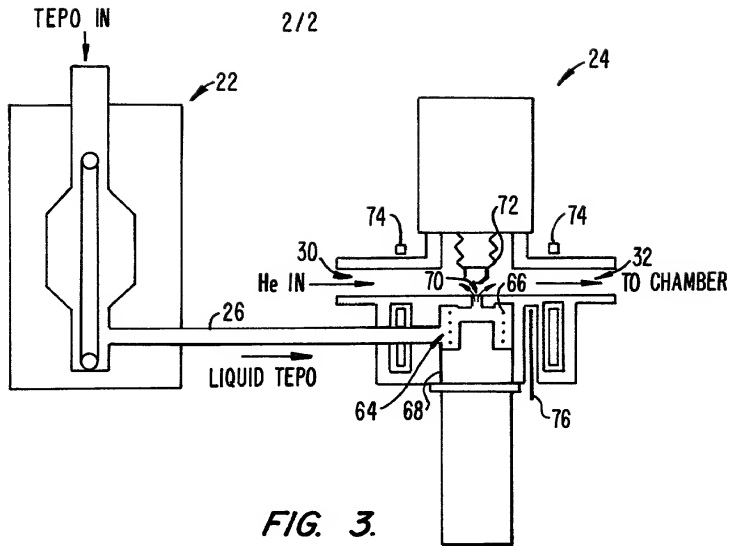


FIG. 2.



COMBINED DECLARATION AND POWER OF ATTORNEY

As a below named inventor, I hereby declare that:

This declaration is of the following type:

- ☒ original
☐ divisional
☐ continuation
☐ continuation-in-part

INVENTORSHIP IDENTIFICATION

My residence, post office address and citizenship are as stated below next to my name. I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

TITLE OF INVENTION

"LIQUID PHOSPHOROUS PRECURSOR DELIVERY APPARATUS"

SPECIFICATION IDENTIFICATION

The specification of which:

- ☒ is attached hereto
☐ was filed on _____, as application Serial No. _____, or
☒ Express Mail No. TB907846610US (as Serial No. not yet known)
and was amended on _____ (if applicable)
☐ was described and claimed in PCT International Application No. _____
filed on _____ and as amended under PCT Article 19 on _____.

ACKNOWLEDGEMENT OF REVIEW OF PAPERS AND DUTY OF CANDOR

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose all information I know to be material to patentability in accordance with Title 37, Code of Federal Regulations, §1.56,

and which is material to the examination of this application; namely, information where there is a substantial likelihood that a reasonable Examiner would consider it important in deciding whether to allow the application to issue as a patent, and

- ☒ In compliance with this duty there is attached an Information Disclosure Statement in accordance with 37 CFR §1.98.

PRIORITY CLAIM (35 U.S.C. §119)

I hereby claim foreign priority benefits under Title 35, United States Code, §119, of any foreign application(s) for patent or inventor's certificate or of any PCT international application(s) designating at least one country other than the United States of America listed below, and have also identified below any foreign application(s) for patent or inventor's certificate or any PCT international application(s) designating at least one country other than the United States of America filed by me on the same subject matter having a filing date before that of the application(s) of which priority is claimed.

☒ No such applications have been filed.

☐ Such applications have been filed as follows:

- A. Prior foreign/PCT application(s) filed within 12 mos. (6 mos. for design) prior to this application, and any priority claims under 35 U.S.C. §119

<u>Country/PCT</u>	<u>Application No</u>	<u>Date Filed</u>	<u>Priority Claimed</u>
			<input type="checkbox"/> Yes <input type="checkbox"/> No
			<input type="checkbox"/> Yes <input type="checkbox"/> No
			<input type="checkbox"/> Yes <input type="checkbox"/> No

- B. All foreign application(s), if any, filed more than 12 mos. (6 mos for design) prior to this U.S. application

Country:
Application No:
Filing date:

PRIOR U.S. APPLICATION(S) FOR WHICH BENEFIT
UNDER 35 U.S.C. §120 IS CLAIMED

<u>Serial No.</u>	<u>Filing Date</u>	<u>Status</u>		
		<u>Patented</u>	<u>Pending</u>	<u>Abandoned</u>
(None)				

POWER OF ATTORNEY

I hereby appoint the following attorneys and/or agents to prosecute this application and transact all business in the Patent and Trademark Office connected therewith:

Lawrence Edelman	Registration No. 25,226
Michael B. Einschlag	Registration No. 29,301
Peter J. Sgarbossa	Registration No. 25,610
Michael L. Sherrard	Registration No. 28,041
Donald Verplancken	Registration No. 33,217
Paul C. Haughey	Registration No. 31,836

Send correspondence to:

Direct telephone calls to:

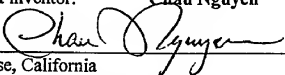
Michael B. Einschlag, MS/2634
Legal Affairs Dept.
Applied Materials, Inc.
3050 Bowers Avenue
Santa Clara, CA 95054.

Paul C. Haughey
(415) 326-2400

DECLARATION

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and, further, that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Sec. 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patents issued thereon.

Full name of sole or first inventor: **Chau Nguyen**

Inventor's signature: 

Date: 12/4/95

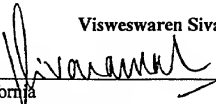
Residence: San Jose, California

Post Office Address: 1748 Morril Avenue
San Jose, CA 95132

Country of Citizenship: U.S.A.

Full name of second inventor:

Visweswaren Sivaramakrishnan

Inventor's signature: 

Date: 12/1/95

Residence: Cupertino, California

Post Office Address: 20303 North Cove Square
Cupertino, CA 95014

Country of Citizenship: India

(Declaration ends with this page)

00700000 111299 002111 19000000